# Ionic Liquids Enabling Revolutionary Closed-Loop Life Support

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Minimizing resupply from Earth is essential for future long duration manned missions. The current oxygen recovery system aboard the International Space Station is capable of recovering approximately 50% of the oxygen from metabolic carbon dioxide. For long duration manned missions, a minimum of 75% oxygen recovery is targeted with a goal of greater than 90%. Theoretically, the Bosch process can recover 100% of oxygen, making it a promising technology for oxygen recovery for long duration missions. However, the Bosch process produces elemental carbon which ultimately fouls the catalyst. Once the catalyst performance is compromised, it must be replaced resulting in undesired resupply mass. Based on the performance of a Bosch system designed by NASA in the 1990's, a three year Martian mission would require approximately 1315 kg (2850 lbs) of catalyst resupply. It may be possible to eliminate catalyst resupply with a fully regenerable system using an Ionic Liquid (IL)-based Bosch system. In 2016, we reported the feasibility of using ILs to produce an iron catalyst on a copper substrate and to regenerate the iron catalyst by extracting the iron from the copper substrate and product carbon. Additionally, we described a basic system concept for an IL-based Bosch. Here we report the results of efforts to scale catalyst preparation, catalyst regeneration, and to scale the carbon formation processing rate of a single reactor.

#### **Nomenclature**

AR= Atmosphere Revitalization = Hydrogen  $H_2$ = Water C= Carbon  $H_2O$ = Ionic Liquid CO= Carbon Monoxide IL= Carbon Dioxide ISS = International Space Station  $CO_2$ = CO<sub>2</sub> Reduction Catalyst Test Stand = Standard milliLiters per Minute COR-CaTS **SmLPM** Cu = Copper

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## I. Introduction

OR long duration life support missions, life support systems must be highly reliable, robust against environmentally-induced failures, and require minimum resupply and crew interaction. Oxygen is a key consumable for crew health. Oxygen aboard the International Space Station (ISS) is provided by the electrolysis of water. Currently, the ISS oxygen recovery system is capable of recovering approximately 50% of the oxygen from metabolic carbon dioxide. For long duration manned missions, a minimum of 75% with a target of 90% of oxygen recovery is required. Theoretically, the Bosch process can recover 100% of oxygen, making it a desirable technology for oxygen recovery for long duration missions. The Bosch process reacts carbon dioxide (CO<sub>2</sub>) with hydrogen (H<sub>2</sub>) to produce water (H<sub>2</sub>O) and elemental carbon (C) in the presence of a catalyst through three reactions:

RWGS	$CO_2 + H_2 \leftrightarrow H_2O + CO$	(1)
CO Hydrogenation	$CO + H_2 \leftrightarrow H_2O + C(s)$	(2)
Boudouard	$2CO \leftrightarrow CO_2 + C(s)$	(3)
Bosch Process	$CO_2 + 2H_2 \leftrightarrow 2H_2O + C(s)$	(4)

As carbon forms on the catalyst, it is chemically dispersed throughout the carbon. Once the catalyst performance is compromised, the catalyst must be replaced resulting in undesired resupply mass. The elimination of catalyst resupply can be achieved with a fully regenerable system. Theoretically, 100% oxygen recovery with zero resupply mass can be achieved with an Ionic Liquid (IL)-based Bosch system. This process involves three major steps, as mentioned previously<sup>1</sup>: electrolysis with an IL to electroplate iron (Fe) onto a copper substrate in a reactor, the Bosch process to convert carbon dioxide to recoverable oxygen and carbon, and catalyst regeneration using the IL to separate the iron catalyst from the solid carbon. When the iron and carbon have been removed, the carbon is then filtered out of the IL and recycled back to the first step of the process. This cycle may be continued indefinitely. Minor handling losses may be made up by extracting *in situ* iron from Lunar or Martian regolith. The success of an IL-based Bosch system will represent a new era of closed loop life support technology that could lean on *in situ* resources as a risk mitigation approach for Lunar or Martian surface missions.

In 2016, a feasibility study of using ILs to reduce catalyst resupply in a closed-loop ECLSS Atmosphere Revitalization (AR) system was conducted and the data gathered suggested such a system to be feasible. Fe was successfully electroplated onto Cu substrates using a traditional method with three ILs. The three ILs used in the study were N-butyl-N-methylpyrrolidinium hydrogen sulfate ([bmpyrr][HSO<sub>4</sub>]), 1-ethyl-3-methylimidazolium hydrogen sulfate ([emim][HSO<sub>4</sub>]), All electroplated Fe Cu substrates were shown to be catalytic from all sources, and Fe extraction from a high carbon-content mixture using [bmpyrr] was demonstrated. Below, we report the results of efforts to scale catalyst preparation, catalyst regeneration, and the carbon formation processing rate of a single reactor.

#### II. Materials and Methods

## A. Materials

All wet chemicals for surface cleaning, IL preparation, and electroplating were purchased from Sigma-Aldrich (St. Louis, MO). Duocel® Cu foam was purchased from ERG Materials and Aerospace (Emeryville, CA) and cut into a 4.6 cm diameter by 1.6 cm thick pucks. Ultra-high purity (UHP) carbon monoxide (CO) gas was purchased from Matheson Tri-Gas (Basking Ridge, NJ). UHP hydrogen and UHP nitrogen gases were purchased from Sexton Welding Supply, Inc. ILs were made at NASA Marshall Space Flight Center.<sup>1</sup>

#### **B.** Scaling of Catalyst Preparation

During initial development efforts, copper substrates were plated individually in a single electroplating cell. A fully integrated system will require multiple substrates to be plated simultaneously. A new design concept, shown in Figure 1, was generated to allow for up to ten substrates to be plated at a time. The newly designed glassware for electroplating consists of two 20.32 cm diameter chambers with a total length of 40.64 cm. The two chambers contain the anode and cathode solutions for the electroplating process. The anode chamber is 20.32 cm in length [A] and contains a hydrogen electrode [B]. The cathode chamber is 20.32 cm in length [C] and contains a rotating disc that secures the copper substrates [D]. The two chambers are identical volume in order for the anode and cathode chamber

to condition the anode to acid. For the batch method, the volume of the cathode electrode returns to acid condition, but this can be controlled by running the chamber to supply hydrogen to the anode and energy through the membrane. The cathode solution as the Fe is plating out supplies power to the anode to bind the H back to the backbone. A balance of material on each side is needed to fuel the reactions. Once the iron is plated out, the anode side is complete and ready to digest more Fe for plating. The cathode side is now ready to go to anode side for the recharge of the hydrogen. If different volumes in the batch method are used, all the iron may not be out of cathode side before the anode is fully recharged. With different volume chambers, the plates may not be covered properly to run the chamber to completion of the side with the low volume of IL. It is important to note that this is really a three volume process. Anode going to acid, cathode being cleaned of Fe, and a third volume

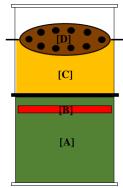


Figure 1. Scaling of Catalyst Preparation
Design. The design concept that will be used to scale the preparation of catalyst from one puck to up to ten pucks simultaneously.

Fe being digested to make a plating solution for pucks. The volumes have to be balanced to keep the electrodes covered for the chamber to run. The anode recharges the acid side and the cathode plates the pucks therefore Fe is being removed. Once each side done, the third chamber is used to keep the reaction re- cycling. Different volume chambers may be used, but one side would have to be reload to get the volume of interest to be fully cleaned or recharged.

The chamber has valves on both sides to permit filling and venting without requiring removal of the end caps. During the electroplating process, the anode chamber is run with a slow flow of hydrogen (7 mL/min at 1 atm) and the cathode chamber will be purged with argon (~7 mL/min). A voltage is applied to the chamber and the copper substrates are plated on one side. After one side of the substrate is plated, the additional length in the cathode chamber allows the disc to be rotated to face the anode plate such that the other side of the substrate is then plated. Unlike the previous design where the anode and cathode solutions were drained to remove the plated substrate, fluids in the newly designed chamber do not have to be removed. The end cap is simply removed and the substrates are unsnapped from the holding disc. Because the chamber is not drained, opened, and cleaned between each run, IL loss between each run is greatly reduced. The design also permits residual IL on the pucks to drain back into the cathode liquid while still under argon purge, preventing the iron from oxidizing. The power requirement is proportional to the surface area being plated.

#### C. Scaling Catalyst Regeneration

Previous regeneration efforts for carbon-coated iron-plated copper substrates have involved regeneration of a single catalyst substrate at a time. In an integrated life support system, an entire reactor including multiple catalyst substrates must be regenerated simultaneously. Carbon deposition on multiple catalyst substrates was performed in order to determine the correlation between mass of deposited carbon and regeneration time. The correlation is then used to determine the reactor size necessary to maximize carbon formation while minimizing regeneration time. Carbon deposition of Fe-plated copper substrates was conducted using the CO<sub>2</sub> Reduction Catalyst Test Stand (COR-CaTS). Prior to testing, each Fe-Cu catalyst puck along with an unplated control Cu puck was placed into a 46mm inner diameter quartz tube reactor along with Unifrax Fiberfrax S insulation discs installed at the inlet side of the control puck and outlet side of the catalyst puck. The insulation discs held the Cu substrates in place and allowed for even distribution of gas flow. The quartz tube was then placed into the Thermolyne F21100 tube furnace. Prior to testing, the system was purged with 400 standard millimeters per minute (SmLPM) of nitrogen. Once the purge was complete, the reactor tubes were heated to 500°C and reduced under a constant stream containing 250 SmLPM of N<sub>2</sub> and 250 SmLPM of H<sub>2</sub>. When the reactor reached temperature, the reactor was fed 300 SmLPM CO, 30 SmLPM H<sub>2</sub>, and 20 SmLPM of N<sub>2</sub>. For each run, when maximum CO<sub>2</sub> was reached, the run was continued for specific durations in order to achieve varied quantities of carbon deposited onto each puck. Upon test completion, 50 SmLPM of N2 was fed to the reactor until the reactor cooled to room temperature. After which, the catalysts were removed from the reactor, weighed to determine total carbon produced, and prepared for regeneration.

During the regeneration process, the carbon-coated Fe-plated puck was placed in a 1.0 mol/L aqueous solution of IL acid of sufficient volume to ensure complete submergence of the pucks (approximately 200mL). The solution was heated to vigorous reflux for 12 to 24 hours using a condenser to capture the water and allowing it to drip back into

the solution under an inert atmosphere (argon). Vigorously refluxing the solution forms nucleate bubbles on the surface of the copper pucks such that the mechanical force helps to dislodge the carbon particles from the puck when they form and burst. During the course of this process, the Fe metal that was plated onto the pucks reacts with the IL acid and dissolves forming  $Fe^{+2}$  sulfate and liberating hydrogen gas, thus extracting the Fe catalyst out of the carbon product and Cu substrate. The purpose of the inert atmosphere is to prevent further oxidation of the  $Fe^{+2}$  to  $Fe^{+3}$ . The carbon particles do not dissolve in or react with the IL solution; however the IL does aid in suspension of the carbon particles. After the puck was clean of carbon, the puck was then ready to be replated. Prior to electroplating, the puck was dipped into a nitiric acid bath to reduce the surface and put into a vaccum oven to dry. The puck was then electroplated and carbon deposition testing was performed on the replated puck using the same process described above.

#### D. Scaling of Carbon Formation Processing Rate of a Single Reactor

Data currently exists for carbon formation rates over single catalyst pucks under specific conditions designed to maximize carbon formation. In order to predict performance in an integrated life support system and to design and accurately scale a prototype system, kinetic data is required. In order to ensure the tests are kinetically limited rather than diffusion limited, a check of flow rate versus reaction rate was completed. This was done by varying the total gas flow rate while keeping the partial pressures of the gases the same. It was determined that the reaction of kinetically limited when there was no longer an increase in conversion as a function of increased flow. To determine kinetic limitation, the Fe-Cu catalyst puck along with an unplated control Cu puck were placed into a 46mm inner diameter quartz tube reactor along with Unifrax Fiberfrax S insulation discs installed at the inlet side of the raw puck and outlet side of the catalyst puck. In order to determine if the reaction was kinetically limited, a flow larger than 1,000 SmLPM of CO was required. Higher capacity mass flow controllers were installed into the COR-CaTS, which controlled gas feeds for CO and H<sub>2</sub> at a range of 10 to 20,000 SmLPM and controlled gas feeds for N<sub>2</sub> at a range of 10 to 2,000 SmLPM. Prior to testing, the system was purged with 400 SmLPM of nitrogen. Once the purge was complete, the reactor tubes were heated to 500°C under 250 SmLPM of N2 and 250 SmLPM of H2. When the reactor reached temperature, a flow sweep was completed with the total gas flow rate varied for CO from 200 SmLPM to 1,250 SmLPM at a composition of 50% CO, 40% N<sub>2</sub>, and 10% H<sub>2</sub> as well as a composition of 50% CO, 10% N<sub>2</sub>, and 40% H<sub>2</sub>. After the tests were complete, a 50 SmLPM N<sub>2</sub> purge was used to cool the reactor to room temperature.

Once it was determined what flow range was required in order to be reaction rate limited, kinetics testing was conducted for the Hydrogenation and Boudouard reaction. The tubes were packed using the same process as previously described, the system was purged with nitrogen, and the catalyst was reduced with a steady flow of 250 SmLPM of  $H_2$  and  $N_2$  until the desired temperature was reached. For the kinetics testing, a total flow rate of 2,000 SmLPM was used for the Hydrogenation reaction and a total flow rate of 1,000 SmLPM was used for the Boudouard reaction with temperatures varying from 250°C to 550°C.

## E. Catalyst Analysis

During the electroplating process, there was a concern that increasing the Fe plating on the substrate decreases the surface area due to the Fe filling pores in the Cu substrate. Fe coverage is a function of axial distance into the substrate and possibly radial distance from the center of the substrate. Three Fe-plated Cu substrates and three non-plated Cu substrates were sent to Reaction Systems, Inc. in Golden, CO for Brunauer-Emmett-Teller (BET) analysis with the intention of cutting samples from identical locations from each substrates to determine an average specific surface area of each location. The BET analysis was performed on the Cu substrates using the single point BET method. A mixture of 30%  $N_2$  in helium (He) was applied over the sample. The cell was then immersed in a Dewar containing liquid  $N_2$ , allowing the sample to cool. Once the sample reached liquid  $N_2$  temperature,  $N_2$  was absorbed by the sample and the change in  $N_2$  concentration was detected and measured by a thermal detector.

## III. Results

## A. Scaling of Catalyst Regneration

Carbon formation was varied on multiple catalyst pucks in order to determine the correlation between mass of deposited carbon and regeneration time. In order to achieve various quantities of deposited carbon onto each catalyst puck, after maximum CO<sub>2</sub> was reached, the runs were continued for various lengths of time. The results of carbon formation on each catalyst puck are shown in Table 1. Prior regeneration efforts were carried out on carbon coated pucks with small amounts of carbon. Regeneration of the carbon-coated pucks with much denser carbon proved to be very challenging. After carbon formation, the pucks had large quantities of carbon deposits both on the outside

surface of the pucks and on the interior cells. The loose carbon on the exterior of the pucks comes off readily and typically falls off upon handling. However removal of the carbon trapped inside the cells was more challenging.

Table 1. Regeneration data for	r Fe-plated copper i	pucks with varied	l amounts of carbon.

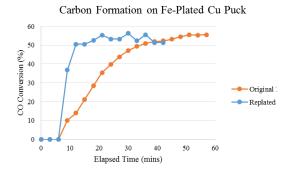
Fe-Plated Cu Substrate	Mass of Fe Plated (g)	Duration after max. CO <sub>2</sub> (min)	Mass of Carbon (g)
1	0.2970	15	2.50
2	0.4521	30	3.78
3	0.2719	60	4.07
4	0.3835	120	3.71

Based on initial experiments it was found that the regeneration process works well for recovering the majority of the Fe; however the carbon deposits (possibly containing some Fe) in the interior cells of the pucks have proven to be quite recalcitrant. Figure 2 shows the carbon that was dropped from a regenerated puck after the puck was dipped in a nitric acid wash and vaccum oven dried prior to replating. By chance, it was found that washing one of the aqueous IL acid treated pucks with acetone removed the vast majority of the interior carbon deposits quite readily. Because acetone is a volatile and flammable organic solvent it is not desirable for use in a space environment; however if acetone can remove the carbon deposits other suitable organic solvents should be able to do the same. Since ILs are technically organic solvents, the plan is to test a highly concentrated aqueous IL acid solution (>90% IL) or even a neat (100%) IL acid and determine the effectiveness for removing the carbon. The resulting suspension of carbon particles in the IL acid can then be diluted with water to a concentration of 1.0 mol/L and heated to react with the iron and convert it to Fe<sup>+2</sup>. The total regeneration time for the pucks were 1 to 2 days.

The regeneration time was longer than required to react with the Fe and solubilize it as Fe<sup>+2</sup>. The reason for such long regeneration times was to help remove more carbon from the pucks, which was determined to be not feasible. Future regeneration shall take less time, particularly if the treatment with the neat IL proves feasible. Since acetone removed carbon from the Cu puck in 10 to 15 minutes, the neat IL should be capable of removing the carbon from the pucks in less than 30 minutes. It takes 1 to 2 hours for Fe to solubilize in the hot aqueous IL acid. Total regeneration time with the neat IL would be 3 to 4 hours at most.

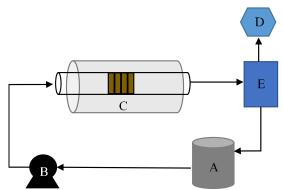
Afer the regeneration process, iron was then replated onto a regenerated puck and carbon deposition testing was performed with the same conditions as the original plated puck. Figure 2 shows the regenerated copper puck after electroplating. Post carbon formation results proved the feasibility of the regeneration process for the cleaning and plating of copper substrates. Figure 3 shows the outlet  $CO_2$  mole percent versus the time for

Figure 2. Fe-plated Cu puck post carbon regeneration (above) and the regenerated Fe-plated Cu puck after electroplating (below).



**Figure 3. CO Conversion versus Elapsed Time.** A graph of the CO conversion versus elapsed time for the original Fe-plated Cu puck and the replated Cu puck.

each puck. Both pucks demonstrated 56% single-pass conversion of CO.



**Figure 4. Bulk Regeneration Design.** The design concept for future bulk catalyst regeneration testing. Carbon coated catalyst pucks inside a quartz tube will then be placed into the reactor (C). The IL in the reservoir (A) will be pumped through the quartz tube. The IL is then run through a filter housing unit allowing for carbon to be filtered out of the IL (E), and the IL then goes into the reservoir where it then will continue the process.  $H_2$  emitted during the process will be collected in a metal hydride container (D).

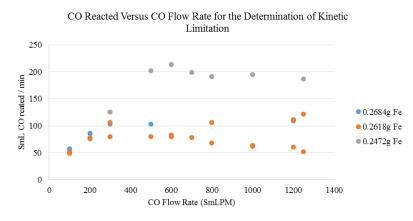
Future regeneration efforts will involve the demonstration of a simplified continuous flow regeneration system in which multiple carbon coated catalyst pucks will be regenerated simultaneously. Figure 4 shows the initial design that will be used in the bulk regeneration demonstration. For bulk regeneration testing, carbon will be deposited onto the Fe-plated Cu pucks using the COR-CaTS. The quartz tube containing the carbon coated catalyst pucks will then be placed into the reactor (C). Once the pucks are secured in the reactor, the IL in the reservoir (A) will be pumped through the quartz tube. The IL is then run through a filter housing unit allowing for carbon to be filtered out of the IL (E), and the IL then goes into the reservoir where it then will continue the process.  $H_2$  emitted during the process will be collected in a metal hydride container (D).

## B. Scaling of Carbon Formation Processing Rate of a Single Reactor

#### 1. Determination of Kinetic Limitation

Kinetic data was required in order to predict performance and accurately scale a prototype in an integrated life support system. A check of flow rate versus reaction rate was completed to determine the flow range required in order to be kinetically limited. The flow sweep was completed at 500°C with the total gas flow rate varied for CO from 200 SmLPM to 1,250 SmLPM at a composition of 50% CO, 40% N<sub>2</sub>, and 10% H<sub>2</sub> as well as a composition of 50% CO,

40% H<sub>2</sub>, and 10% N<sub>2</sub>. Since all runs were at CO pp of 7.5 psia, a plot of CO reacted per minute versus CO flow rate for all compositions is shown in Figure 5. Three catalyst pucks with varied amounts of Fe deposited onto the surface were used to obtain all data points. The variation in the data shown in Figure 5 is due to the differences in surface area caused by the amount of Fe depositied onto each catalyst puck. Based on the experimental data it, was determined that a CO flow rate between 500 to 1,000 SmLPM was needed in order to be considered kinetically limited.



**Figure 5.** A graph of CO reaction rate for all runs with ppCO of 7.5psi and the partial pressure held at a constant 7.5psi.

## 2. Hydrogenation Reaction Kinetic Data

Carbon monoxide and H<sub>2</sub> react to form H<sub>2</sub>O and solid carbon for the Hydrogenation reaction, as shown in Equation

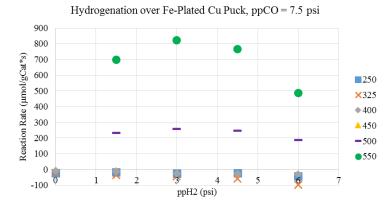
2. There are two proposed kinetic models used to describe the hydrogenation of CO to form solid carbon.<sup>2,3</sup>

$$r_c = k_5 P_{CO}{}^a P_{H_2}{}^b (5)$$

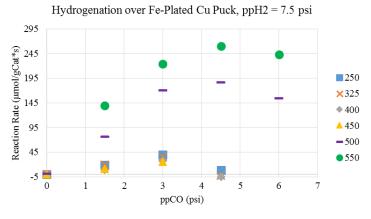
$$r_c = k_6 (P_{CO} P_{H_2})^h (6)$$

For Hydrogenation testing, the Fe-plated Cu puck was presented with CO and  $H_2$  in the feed stream. The Hydrogenation reaction rates at various temperatures over Fe-plated Cu substrates is shown in Figure 6. Activity toward catalyzing the hydrogenation reaction of Fe-plated copper substrates was shown at temperatures of 500°C and above when CO was held constant. When  $H_2$  was held constant, activity toward catalyzing the hydrogenation reaction occurs at temperatures 250°C and above (see Figure 7).

The determined hydrogenation reaction rates and the partial pressures of the  $H_2$  and CO were used to evaluate a, b, h, and the activation energies from the equations 5 and 6 above. The determined values for each are shown in Table 2. A plot of the predicted versus experimental reaction rates shown in Figure 8 shows that the experimental data does not correspond with the predicted values from Equation 5. The poor fit is likely due to the inability to control the competing Boudouard and Sabatier reactions occurring during the Hydrogenation reaction.



**Figure 6. Hydrogenation on Fe-Plated Cu Pucks.** Hydrogenation rate versus the inlet partial pressure of  $H_2$  with the inlet CO partial pressure held at a constant 7.5psi.



**Figure 7. Hydrogenation on Fe-Plated Cu Pucks.** *Hydrogenation rate* versus the inlet partial pressure of CO with the inlet H<sub>2</sub> partial pressure held at a constant 7.5psi.

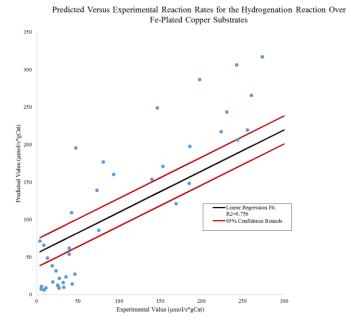


Figure 8. Predicted versus Experimental Reaction Rates for Hydrogenation over Fe-Plated Copper Substrates. The predicted Hydrogenation reaction rate versus the experimental reaction rate for equation 5.

**Table 2. Calculated Hydrogenation Rate Equation Constants.** Rate equation constants determined from experimental data.

Calculated Hydrogen Constants		
a	0.4985	
b	-0.1501	
h	0.5321	
$E_5$	69.70 kJ/mol	
$E_6$	23.98 kJ/mol	

## 3. Boudouard Reaction Kinetic Data

Carbon monoxide reacts to form  $CO_2$  and solid carbon in the Boudouard reaction as seen in equation 3. The Boudouard reaction kinetic data was collected for Fe-Cu pucks with only CO as the reactant in the feed stream at temperatures from 250°C to 550°C. There were two kinetic models used to explain the reaction over the catalyst at various temperatures and pressures.<sup>2</sup>

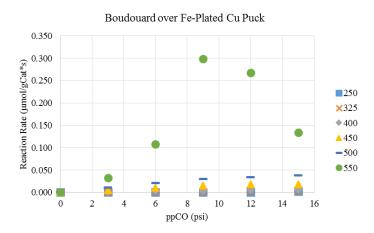
$$r_{c} = k \frac{P_{CO}}{\left[1 + K_{A} P_{CO} + K_{B} \left(\frac{P_{CO_{2}}}{P_{CO}}\right)\right]^{2}}$$
 (7)

$$r_c = k \frac{P_{CO}^2}{[1 + K_A P_{CO}]^2} \tag{8}$$

Since no CO<sub>2</sub> was introduced into the Boudouard feed stream for testing, Equation 7 was modified to the following:

$$\sqrt{\frac{P_{CO}}{r_c}} = \frac{1}{\sqrt{k}} + \frac{K_A}{\sqrt{k}} P_{CO} \tag{9}$$

In order to determine the values for K<sub>A</sub> and k, a graph of the left side of Equation 9 versus the partial pressure of CO was used. The Boudouard reaction rates at various temperatures over Fe-plated Cu substrates is shown in Figure 9. Activity toward catalyzing the Boudouard reaction of Feplated copper substrates was shown at temperatures of 400°C and above. Based on the fit of the data shown in Figure 10, both models accurately predict the reaction rates. The activation energies for both models are shown in Table 3. The activation energy of Model 2 is close to the reported activation energy for iron at temperatures below 550°C by Westerman (83.68kJ/mol).



**Figure 9. Boudouard Reaction over Fe-Plated Cu Pucks.** Boudouard reaction rate versus the inlet partial pressure of CO.

Predicted verses Experimental Reaction Rates for the Boudouard Reaction over Fe-Plated Cu Substrates Using Models 1 and 2

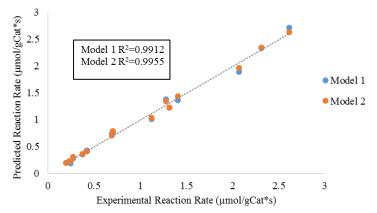


Figure 10. Predicted versus Experimental Reaction Rates for Boudouard Model 1 and 2. A plot of Model 1 and Model 2 predicted versus experimental reaction rates.

Table 3. Calculated Activation Energy for Boudouard Reaction. Activation energies determined from experimental data.

Calculated Hydrogen Constants		
$E_5$	100.64 kJ/mol	
E <sub>6</sub>	84.49 kJ/mol	

### C. Catalyst Analysis

Three Fe-plated Cu substrates and three non-plated Cu substrates were sent to Reaction Systems, Inc. in Golden, CO for BET analysis with the intention of cutting samples from identical locations from each substrates to determine an average specific surface area of each location. Due to having to process approximately half of each substrate in order to achieve a detection limit, it was not possible to determine surface areas at specific locations on the substrate. The values of the surface areas of each substrate determined by Reaction Systems, Inc. are shown in Table 4. Substrate 3 absorbed more N<sub>2</sub> than the other substrates. Based on substrate 2 and 4, it was concluded that surface area decreases with increasing the amount of Fe deposited on the surface as suspected.

**Table 4. BET Surface Area Analysis of Copper Substrates.** *The BET surface area analysis results of non-*plated copper substrate and three plated copper substrates with various amounts of Fe plated.

Substrate	Amount of Fe (g)	Surface Area (m²/g)
1	0.000	0.038
2	0.255	0.062
3	0.305	0.580
4	0.634	0.022

## Acknowledgments

The authors would like to thank Heath Mullins for test stand support, Tom Williams for software support, and Reaction Systems, Inc. for BET analysis.

#### **Conclusion and Future Work**

An IL-based Bosch system approach will provide a fully regenerable technique for recovering oxygen from either metabolic CO2 or from atmospheric CO2 utilizing ILs and *in situ* resources. Past studies proved the feasibility of such system and provided a first generation IL-based Bosch system concept. Fe was successfully electroplated onto Cu substrates, all electroplated Cu substrates were shown to be catalytic from all sources, and Fe extraction from a high carbon-content mixture using an IL was demonstrated. The data reported here shows efforts to scale catalyst preparation, scale catalyst regeneration, and scale the carbon formation processing rate of a single reactor. A new design concept for plating multiple catalyst substrates was presented. Carbon formation was completed on multiple catalyst pucks in order to determine the correlation between mass of deposited carbon and regeneration time. Regeneration of the carbon-coated substrates proved to be challenging and efforts to reduce regeneration time are being investigated. Kinetic data was collected to help predict performance in an integrated life support system and to design and accurately scale a prototype system. Future work will include demonstration of the new design chamber for electroplating multiple catalyst simultaneously, demonstration of a simplified continuous flow regeneration system, and a second generation concept and initial reactor design will be generated for the IL-based Bosch process.

#### References

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